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## Strength and thermal stability of Cu-Al<sub>2</sub>O<sub>3</sub> composite obtained by internal oxidation<sup>(\*)</sup>

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### Abstract

The objective of the work is to study the effects of the high-energy milling on strengthening, thermal stability and electrical conductivity of Cu-Al<sub>2</sub>O<sub>3</sub> composite. The prealloyed copper powders, atomized in inert gas and containing 3 wt. % Al, were milled up to 20 h in the planetary ball mill to oxidize *in situ* aluminium with oxygen from the air. Composite compacts were obtained by hot-pressing in an argon atmosphere at 800 °C for 3 h under the pressure of 35 MPa. The microstructural characterization was performed by the optical microscope, scanning electron microscope (SEM), transmission electron microscope (TEM) and X-ray diffraction analysis (XRD). The microhardness, electrical conductivity and density measurements were also carried out. The effect of internal oxidation and high-energy milling on strengthening of Cu-Al<sub>2</sub>O<sub>3</sub> composite was significant. The increase of the microhardness of composite compacts (292 HV) is almost threefold comparing to compacts processed from the as-received Cu-3 wt. % Al powder (102 HV). The grain size of Cu-Al<sub>2</sub>O<sub>3</sub> compacts processed from 5 and 20 h-milled powders was 75 and 45 nm, respectively. The small increase in the grain size and the small microhardness drop indicate the high thermal stability of Cu-Al<sub>2</sub>O<sub>3</sub> composite during high-temperature exposure at 800 °C.

### Keywords

Powder processing; Metal matrix composites; Microstructural characterization; Microhardness; High-temperature properties.

## Resistencia y estabilidad térmica del compuesto Cu-Al<sub>2</sub>O<sub>3</sub> obtenido con oxidación interna

### Resumen

El objetivo del trabajo es el estudio de los efectos de la pulverización con altas energías sobre la resistencia, estabilidad térmica y conductividad eléctrica del compuesto Cu-Al<sub>2</sub>O<sub>3</sub>. El polvo pre-aleado de cobre, obtenido a través de la atomización con gas inerte y con un contenido de 3wt. % Al, se molió durante 20 h en el molino planetario de bolas dando lugar a la oxidación *in situ* del aluminio con el oxígeno del aire. El compuesto compactado se ha obtenido mediante prensado en caliente en atmósfera de argón a 800 °C durante 3 h y a una presión de 35 MPa. La caracterización microestructural se hizo a través de microscopía óptica, microscopía electrónica de barrido (SEM), microscopía electrónica de transmisión (TEM) y difracción de rayos X (XRD). También se realizaron medidas de micro-dureza, de conductividad eléctrica y de densidad. La micro-dureza de los compuestos compactados (102 HV) aumentó casi tres veces comparada con la de los obtenidos del polvo Cu-3 wt. % Al (102 HV). El tamaño de grano del compactado Cu-3 wt. % Al fue 75 y 45 nm después de 5 y 20 h de pulverización, respectivamente. El pequeño aumento del tamaño de grano y la pequeña caída de micro-dureza del compuesto Al<sub>2</sub>O<sub>3</sub> indican alta estabilidad térmica durante la exposición a altas temperaturas de 800 °C.

### Palabras claves

Procesado de polvo; Compuestos metal-matriz; Caracterización microestructural; Microdureza; Propiedades a altas temperaturas.

## 1. INTRODUCTION

Oxide dispersion strengthened (ODS) copper matrix have been extensively studied in recent years due to attained better properties than pure copper and copper alloys reinforced by precipitation and solid

solution hardening. ODS offers a unique combination of high electrical and thermal conductivities with high strength at room and elevated temperatures encouraging research to further improvement of this process. Manufacturing methods involved in obtaining copper-matrix composites by powder

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metallurgy techniques include processes such as: mechanical mixing, mechanical alloying, internal oxidation and coprecipitation. As uniformly distributed particles of nanometric size can be achieved by application of internal oxidation, whereas high-energy milling is used for production of ultrafine grained powders, combination of these two methods has been proved as very common techniques for processing of copper-based composites with a fine dispersion of  $\text{Al}_2\text{O}_3$  particles and small grains, which are retained even after compaction at high temperature<sup>[1]</sup>. Therefore, dispersion hardening and grain refinement strengthening are two mechanisms contributing in improvement of strengthening of these composites. Further, high diffusion rate of oxygen through deformed particles during high-energy milling in air enables internal oxidation of aluminium in prealloyed copper matrix.  $\text{Cu-Al}_2\text{O}_3$  composites obtained by this relatively simple technique possess improved properties compared with other methods. Microhardness of  $\text{Cu-Al}_2\text{O}_3$  composites obtained by high-energy milling in air reached values between 216 and 306 HV<sup>[1 and 2]</sup>, whereas thermal stability expressed as microhardness softening temperature was approximately 800 °C. Although the influence of amount of  $\text{Al}_2\text{O}_3$  must not be neglected, values of microhardness of composites obtained by a synergetic effects of high-energy milling and internal oxidation are superior to those of composites produced either by internal oxidation (between 89 and 155 HV)<sup>[3-6]</sup>, or mechanical alloying (between 79 and 98 HV)<sup>[7 and 8]</sup>. Thermal stability in all these cases is quite high, *i.e.* between 880 and 950 °C<sup>[3-8]</sup>. Applying high-energy milling of a mixture of Cu and  $\text{Al}_2\text{O}_3$  powders a considerably high microhardness (250 HV) has recently been obtained<sup>[9]</sup>, but with a relatively low thermal stability. After exposure at 500 °C for 1h the microhardness of this material decreased sharply to 183 HV.

Generally, the results concerning processing of ODS copper matrix composites reported in the literature are quite inconsistent and the influence of many parameters regarding reinforcing phenomena of these composites still have to be explained.

Applying method of simultaneous internal oxidation and milling in air, very fine dispersion of  $\text{Al}_2\text{O}_3$  and small grain sized microstructure may be obtained. This method is advantageous compared with other methods of composite production when internal oxidation is performed as the first process, whereas the additional deformation is necessary to be carried out in order to obtain a small grain size structure.

Taking into account the importance of the role of  $\text{Al}_2\text{O}_3$  on properties of ODS copper-matrix

composites the effect of internal oxidation during high-energy milling of prealloyed copper powders with 3 wt. % Al on strengthening, thermal stability and electrical conductivity of  $\text{Cu-Al}_2\text{O}_3$  composite was examined in this study.

## 2. EXPERIMENTAL

The prealloyed copper powders, atomized in inert gas and containing 3 wt. % Al, were milled for 3 h in air in the planetary ball mill and then the milling was continued in steps of 5 h up to 20 h. The milling served to obtain a fine grained structure and formation of  $\text{Al}_2\text{O}_3$  dispersoids by internal oxidation. The ratio of powder to steel balls' weight was 1:35. During high-energy milling of prealloyed copper powders, aluminium as being more noble element than copper, is the first that oxidizes through the reaction with oxygen from the air and forms a uniform distribution of nano-sized  $\text{Al}_2\text{O}_3$  particles<sup>[1]</sup>. After milling, all powders were processed for 1 h in hydrogen at 400 °C to eliminate copper oxides. Composite compacts with dimensions  $D = 15$  mm,  $H = 10$  mm were obtained by hot-pressing for 3 h in an argon atmosphere at 800 °C under the pressure of 35 MPa. Composite compacts from 5 - and 20 h-milled powders were annealed for 5 h in argon at 800 °C to examine their thermal and electrical stability. Compacts processed from as-received prealloyed copper and electrolytic copper powders were also prepared under the same conditions as composite compacts.

$\text{Cu-3 wt. \% Al}$  powders and composite were characterized by X-ray diffraction analysis (XRD), optical microscope, scanning electron microscope (SEM) equipped with electron dispersive spectroscopy (EDS), and transmission electron microscope (TEM).

Samples for optical microscope were mounted in acrylic resin. Polishing was performed using the standard procedure and a mixture of 5 g  $\text{FeCl}_3$  and 50 ml  $\text{HCl}$  in 100 ml distilled water was used for etching.

Only a few specimens were investigated with TEM. For TEM characterization, slices (less than 1 mm thick) were cut from the cross-section of the compact. The slices were further thinned to 100  $\mu\text{m}$  by a conventional grinding 3 mm discs were punched and electropolished in a Struers twin jet electropolisher. The electropolishing was performed in the mixture of  $\text{CH}_3\text{OH}$  and  $\text{HNO}_3 = 3:1$ , under the following conditions:  $T = -35$  °C,  $U = 9$  V and  $I = 20$  mA.

The lattice parameters were determined for all tested reflections using the least square roots method. Equation according to Klug and Alexander<sup>[10]</sup> was applied:

$$a_{hkl} = d_{hkl} (h^2 + k^2 + l^2)^{1/2} \quad (1)$$

where  $d$  is interplanar distance, whereas  $h$ ,  $k$  and  $l$  are Miller indices.

The average lattice distortion, *i.e.* the relative deviation of the lattice parameters from their mean value ( $\Delta d/d$ )<sup>[11]</sup> and the grain size ( $D$ ) of Cu-3 wt. % Al powder and compacts were determined from the broadening ( $b$ ) of the first four diffraction lines (111, 200, 220 and 311) using the approach of Williamson and Hall<sup>[12]</sup>:

$$\beta \cos \theta = \frac{k}{D} \lambda + \frac{k \Delta d}{d} \sin \theta \quad (2)$$

where the shape factor  $k$  is 0.9 and radiation wave length  $\lambda$  is 0.15405 nm. In this paper the method separating the broadening of lines connected with the grain size from that due to stresses imposed to the crystal lattice was applied. The influence of anisotropy on the line width has not been considered. Correction of the instrumental broadening was executed<sup>[13]</sup>. In this way, it was possible to obtain the real size of the grains. X-ray diffraction analysis was performed with Cu-K $\alpha$  Ni filtered radiation.

The strengthening of the copper matrix was estimated by microhardness measurement where applied load was 50 g. The electrical conductivity (% IACS, IACS<sub>20°C</sub> = 0.5800  $\mu\Omega^{-1} \text{ cm}^{-1}$ ) of polished

compacts was measured with apparatus operating at 60 KHz with electrode diameter of 14 mm.

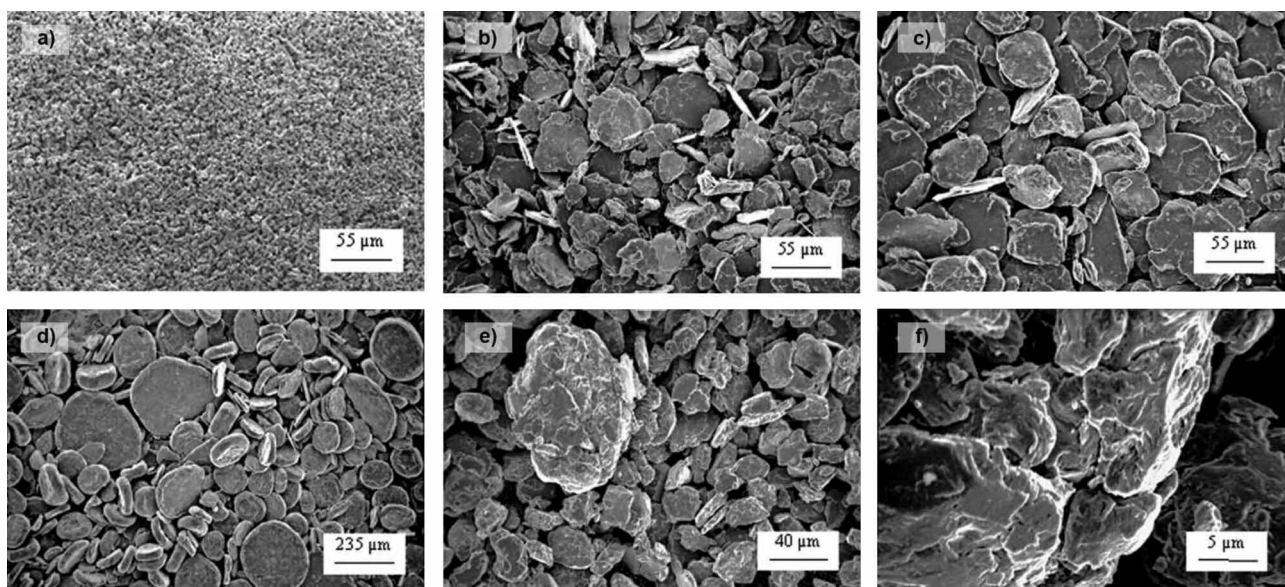
The density of compacts ( $\rho$ ) was determined by the Archimedes method. The theoretical density of compacts was calculated by the simple rule of mixtures assuming 8.96 and 3.95  $\text{g cm}^{-3}$  the fully dense values for copper and Al<sub>2</sub>O<sub>3</sub>, respectively.

Values of density, microhardness and electrical conductivity represent the mean value of five measurements performed on the same compact.

### 3. RESULTS AND DISCUSSION

Generally, during high-energy milling powder particles change their morphology and microstructure as a consequence of repeated deformation, fracturing and welding<sup>[14]</sup>. The change of particles morphology during milling is illustrated in figure 1.

SEM micrograph shows as-received Cu-3 wt. % Al powder, *i.e.* particles before milling (Fig. 1a)) and the development of powder morphology with increasing milling time (Figs. 1b) e)). The particle size increases during up to 5 h of milling due to the predominance of welding. The particles are rather flattened because of the strong plastic deformation in the early stage of the milling (Figs. 1b) and c)). After 10 h of milling, the particle size decreased since the fracturing predominates in the milling process



**Figure 1.** SEM. Morphology of as-received and milled Cu-3 wt.% Al particles: (a) as-received; (b) 3 h; (c) 5 h; (d) 10 h, (e) 20 h, (f) detail of figure 6 e).

*Figura 1. SEM. Morfología de las partículas sin tratar y de las molidas de Cu-3 wt. % Al: (a) sin tratar; (b) 3 h; (c) 5 h; (d) 10 h, (e) 20 h, (f) detalle de la figura 6 e).*



(Fig. 1d)). In the case of balance of fracture and welding, the particles are quite uniform in size and equiaxed in shape. Since during 20 h of milling, the particles appear to be rather uniform in size (Fig. 1e)), but not equiaxed in shape, it is obvious that the balance of fracture and welding processes has not been reached. The presence of some coarse particles (Figs. 1e) and f)) indicates that after 20 h of milling, the coalescence occurs in some extent.

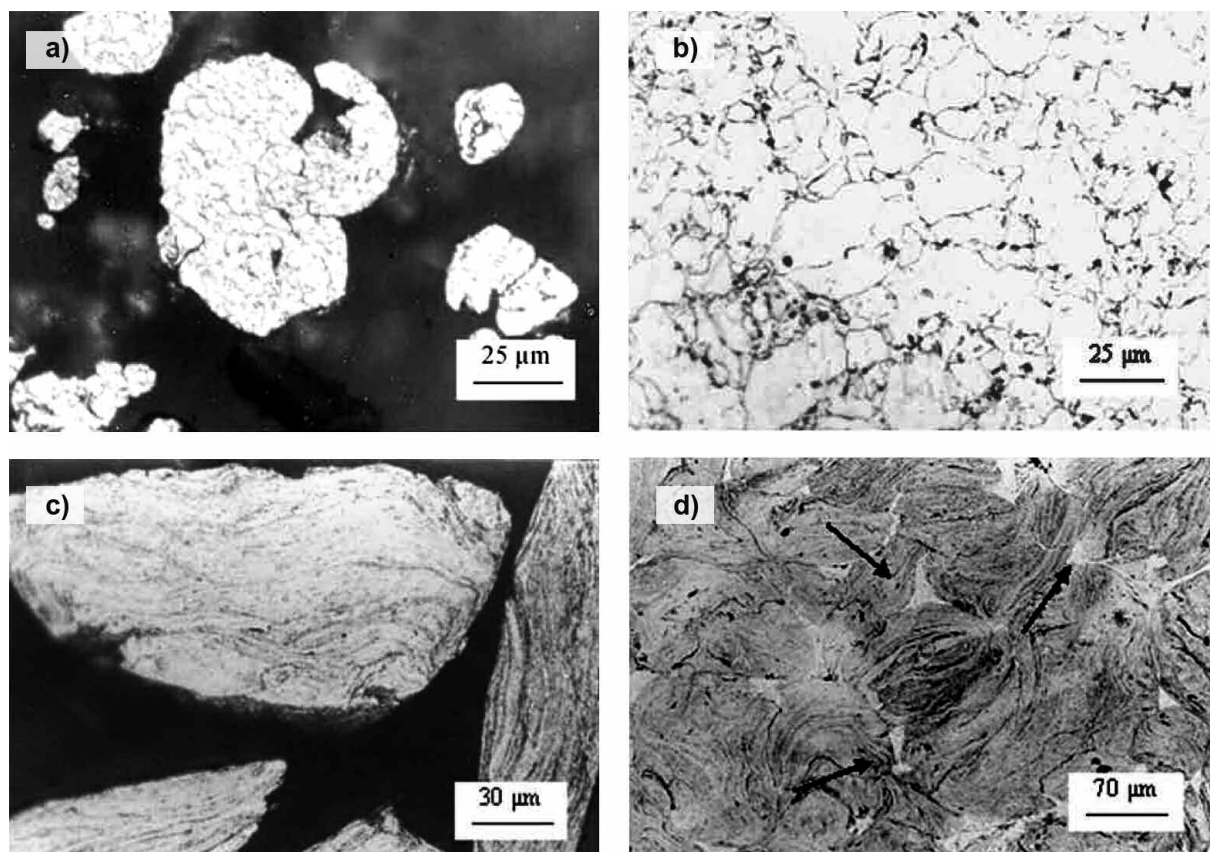
Figure 2 illustrates the microstructure of as-received and 20 h-milled  $\text{Cu-3 wt. \% Al}$  powder particles (Figs. 2a) and c), respectively) and hot-pressed compacts processed from as-received  $\text{Cu-3 wt. \% Al}$  powders and those milled for 20 h (Figs. 2b) and d), respectively).

Comparing the microstructure of the as-received powder (Fig. 2a)) with the milled powder particles (Fig. 2c)), it is evident that milled powder particles exhibit lamellar structure typical for high-energy treated powders, where lamellae represent individual

plastically deformed prealloyed copper particles. Polygonal grains of different size may be seen in the micrograph of hot-pressed compacts processed from as-received  $\text{Cu-3 wt. \% Al}$  powders (Fig. 2b)). On the other side, following hot-pressing the lamellar structure of 20 h-milled powders is retained in the composite (Fig. 2d)). The light areas (denoted by arrows in figure 2d)), indicate that the recrystallization occurred during hot-pressing. The recrystallization was initiated at the corners of the particle powder where the concentration of stresses imposed during compaction was highest.

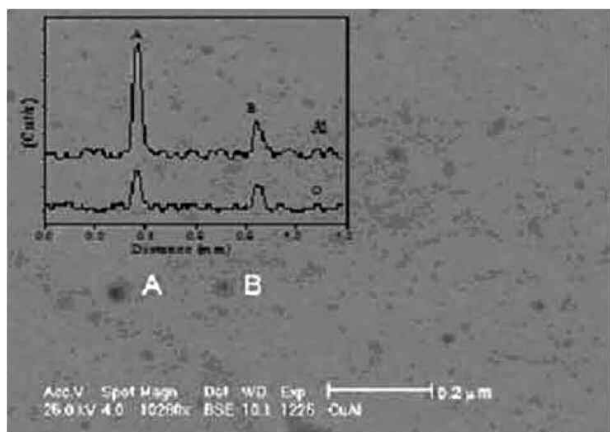
SEM micrograph (Fig. 3) of 20 h-milled compact reveals small particles with the size ranging between 30 and 50 nm in the copper matrix. EDS peaks show the existence of aluminium and oxygen in these particles confirming the presence of  $\text{Al}_2\text{O}_3$ .

The shape of  $\text{Al}_2\text{O}_3$  particles appearing as dark spots in TEM micrograph (Fig. 4) of the same compact is rather irregular with the approximate size



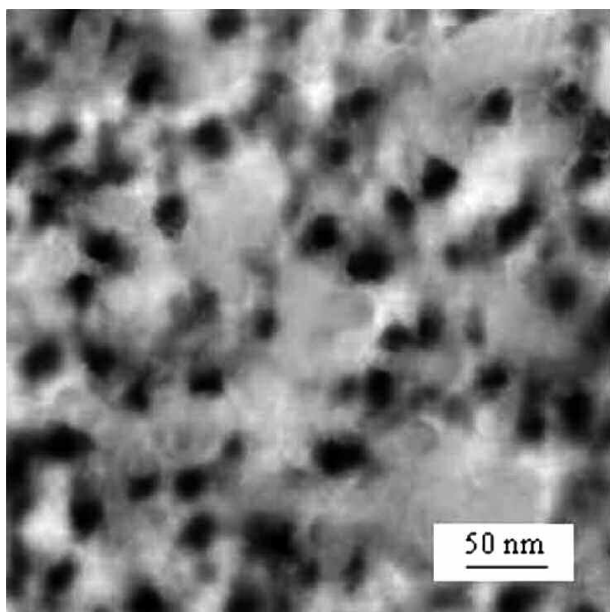
**Figure 2.** Optical microscope. Microstructures of  $\text{Cu-3 wt. \% Al}$  powders and composites. (a) as-received powder; (b) compact processed from as-received powders; (c) 20 h-milled powder and (d) composite compact processed from 20 h-milled powders.

*Figura 2. Microscopía óptica. - Microestructuras de los polvos y de los compuestos de  $\text{Cu-3 wt. \% Al}$ . (a) polvo sin tratar; (b) compuesto procesado del polvo sin tratar; (c) polvo molido 20 h, y (d) compuesto compacto procesado a partir del polvo molido durante 20 h.*



**Figure 3.** SEM micrograph with EDS of 20 h-milled compact.

*Figura 3. Imagen SEM con EDS del compuesto molido 20 h.*

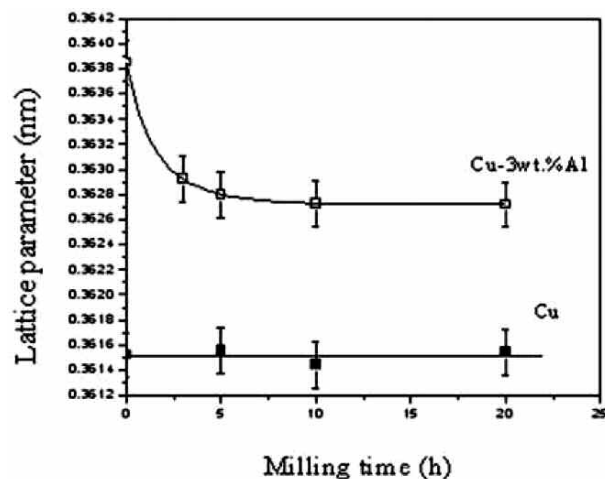


**Figure 4.** TEM micrograph of 20 h-milled compact.

*Figura 4. Imagen TEM del compuesto molido 20 h.*

of 50 nm and less. The grain boundaries could be barely identified. However, supposing that these particles are situated at the grain boundaries then the grain size after 20 h of milling is approximately between 50 and 100 nm.

The relationship between lattice parameters of Cu-3 wt. % Al and electrolytic copper powders vs. milling time is shown in figure 5.



**Figure 5.** Lattice parameters of Cu-3 wt. % Al and electrolytic copper powders vs. milling time.

*Figura 5. Parámetro de red del Cu-3 wt. % Al y de los polvos de cobre electrolítico vs. tiempo de molido.*

The rapid decrease in lattice parameter of prealloyed copper occurs at the very beginning of the milling process. Then the change in lattice parameter decreases slowly with the prolonged milling. It is obvious that discrepancies in lattice parameters between prealloyed copper powders and copper clearly exist. The lattice parameter of prealloyed copper decreases with milling time due to diffusion of  $\text{Al}^{+3}$  and  $\text{O}^{-2}$ , and  $\text{Al}_2\text{O}_3$  dispersoids were formed according to the equation:

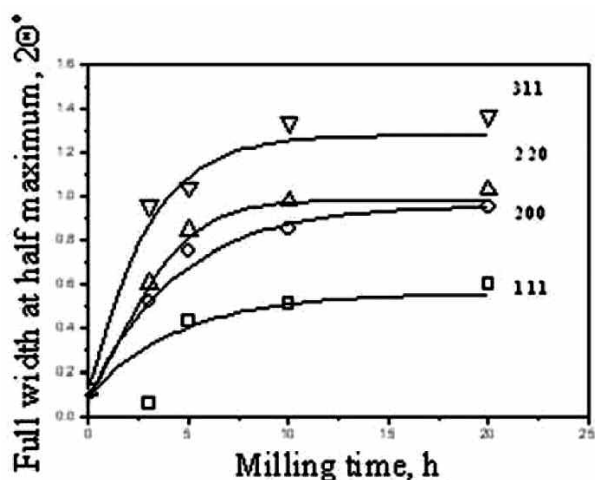


Supposing that the complete amount of aluminium was oxidized, it was calculated that 5.6 wt. % of  $\text{Al}_2\text{O}_3$  was generated in the copper matrix by internal oxidation of prealloyed copper with 3 wt. % Al. The difference in lattice parameters (0.32 %) of prealloyed Cu-3 wt. % Al powders before ( $a_{\text{Cu-3 wt. \% Al}} = 0.36385 \text{ nm}$ ) and after milling ( $a_{\text{Cu-3 wt. \% Al}} = 0.36270 \text{ nm}$ ) is similar to the difference (0.30 %) in theoretical lattice parameters of the prealloyed powder ( $a_{\text{Cu-3 wt. \% Al}} = 0.36260 \text{ nm}$ ) and the copper powder ( $a_{\text{Cu}} = 0.36152 \text{ nm}$ ). This supports the assumption that 5.6 wt. % of  $\text{Al}_2\text{O}_3$  was generated in the copper matrix by internal oxidation of prealloyed copper with 3 wt. % Al. According to this calculation it was assumed that after 20 h of milling aluminium diffused from the copper matrix and the complete amount of aluminium was oxidized. However, there was no the exact experimental evidence whether a total amount of aluminium was oxidized, or a small quantity remained in the solid solution.

In contrast, the lattice parameter of copper powder remained constant which favours the assumption that there was no deformation of the copper lattice due to oxidation.

X-ray diffraction pattern of Cu-3 wt. % Al powder shows a progress in line broadening with the milling time (Fig. 6) as a result of a severe lattice distortion and the grain size refinement<sup>[11 and 13]</sup>.

The effect of milling time on the grain size and lattice distortion of powder particles is presented in figure 7.

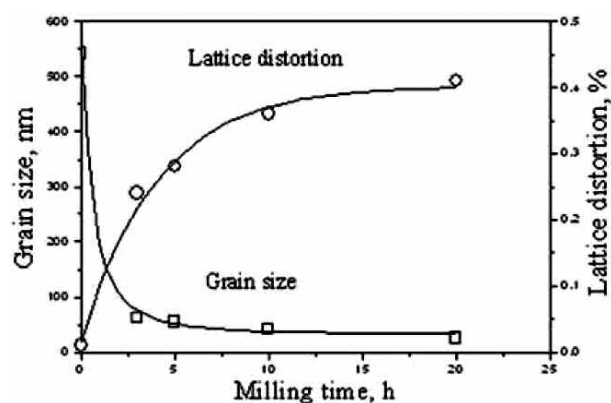


**Figure 6.** Full width at half maximum (FWHM) vs. milling time for Cu-3 wt. % Al powders.

*Figura 6. Anchura a media altura (FWHM) vs. tiempo de molienda para los polvos Cu-3 wt. % Al.*

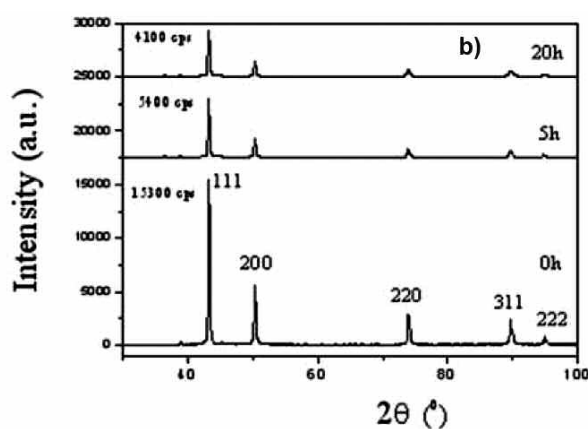
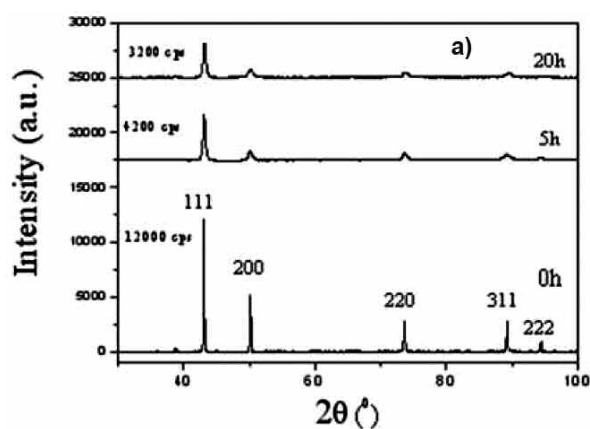
The most intensive grain refinement occurs in the early stage of milling when the grain size abruptly decreases from about 542 to about 55 nm, whereas in the period from 5 h to 20 h the grain size remains practically constant, i.e. approximately 30 nm. Figure 7, also illustrates a strong increase of lattice distortion during 5 h of milling. The lattice distortion becomes less evident at longer milling. This result is in agreement with the hypothesis that the deformation of powder particles occurs during the early stage of milling<sup>[13]</sup>.

The effect of milling time on XRD profiles of Cu-Al<sub>2</sub>O<sub>3</sub> powder and corresponding compacts is illustrated in figure 8.



**Figure 7.** Effect of milling time on grain size and lattice distortion of Cu-3 wt. % Al powders.

*Figura 7. Efecto del tiempo de molienda sobre el tamaño de grano y distorsión de red del polvo Cu-3 wt. % Al.*



**Figure 8.** The effect of milling time on XRD profiles of Cu-Al<sub>2</sub>O<sub>3</sub> powder (a) and corresponding compacts (b).

*Figura 8. Efecto del tiempo de molienda sobre los perfiles XRD del polvo Cu-Al<sub>2</sub>O<sub>3</sub> (a) y los compuestos correspondientes (b).*

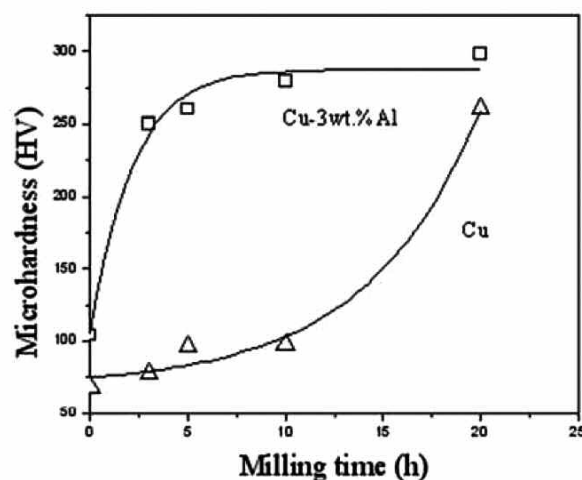


The peak height decreases with the milling time. Although the difference in peak height of powder and compacts is relatively small it may be seen that this height is somewhat higher in the case of compacts suggesting increased grain size as a consequence of diffusion processes during hot-pressing. Applying XRD analysis it was not possible to detect  $\text{Al}_2\text{O}_3$  due to very small powder particles size. The same problem was also mentioned by other authors.

Microhardness of both composite and electrolytic copper compacts increases with the previous milling time (Fig. 9).

The change in microhardness with milling time strongly depends on the composition of compacts. The microhardness of copper compacts is much lower than that of composites, slowly increasing during shorter milling time and reaching higher value at 20 h of milling. On the other side, microhardness of composite increases with the milling time showing that 20 h-milled composite exhibits significantly higher microhardness (up to 292 HV) than compact processed from as-received powders (102 HV). The abrupt increase in microhardness occurs within 5 h of milling while prolonged milling results in a rather small increase in microhardness. The significant decrease in grain size after 5 h of milling (see Fig. 7) strongly suggests that grain size has a very strong effect on strengthening of Cu-3 wt. % Al composite. On the other side, previous investigation<sup>[1]</sup> showed that most of  $\text{Al}_2\text{O}_3$  dispersoids formed by internal oxidation in the copper matrix, were finer than 100 nm and well within the range for dispersion hardening<sup>[15]</sup>. Bearing this in mind and apart from the grain size, the effect of these dispersoids on microhardness cannot be neglected. The effect of content of  $\text{Al}_2\text{O}_3$  dispersoids on strengthening is not quite clear. According to the results published by Mehta *et al.*<sup>[8]</sup> and Nadkarni and Synk<sup>[16]</sup>  $\text{Al}_2\text{O}_3$  content above 0.65 wt. % did not result in increase of strengthening.

The microhardness of composite obtained from 5 h-milled powder with the grain size of about 55 nm is almost 4 times higher (255 HV) than microhardness of compact processed from as-received electrolytic copper powder (67 HV) compacted under the same conditions. The stability of microhardness with prolonged milling time suggests that small grain size is the main parameter influencing the process of strengthening. The coarsening of  $\text{Al}_2\text{O}_3$  dispersoids during milling should have lowered the microhardness. However, because in this study microhardness remains constant during up to 20 h of milling, it may be assumed that the coarsening of  $\text{Al}_2\text{O}_3$  dispersoids did not occur.



**Figure 9.** The effect of milling time on microhardness of Cu- $\text{Al}_2\text{O}_3$  and electrolytic copper compacts.

*Figura 9. El efecto del tiempo de molienda sobre la microdureza del Cu- $\text{Al}_2\text{O}_3$  y los compuestos de cobre electrolítico.*

The effects of the high-temperature exposure (at 800 °C for 5 h) on the grain size and microhardness of composite compacts processed from 5 - and 20 h-milled powders and electrolytic copper are presented in table I. Although statistically insufficiently documented, it should be noted that TEM result concerning the grain size (see Fig. 4) is in fairly good agreement with the calculated grain size of 20 h-milled compact before exposure at 800 °C.

In general, Cu- $\text{Al}_2\text{O}_3$  composite is characterized by low increase in the grain size and by low decrease in the microhardness. This behaviour is a consequence of the presence of very fine  $\text{Al}_2\text{O}_3$  particles acting as a barrier to the increase of grains under the high temperature.

Results from table I indicate that the increase in the grain size of Cu- $\text{Al}_2\text{O}_3$  composite after high-temperature exposure is rather small (only about 8 %) compared to the initial grain size (before high-temperature exposure). Changes in the grain size are in accordance with the difference in the peak heights in XRD (Fig. 10), *i.e.* after high temperature exposure peaks are somewhat higher indicating the increase of the grain size.

This increase may be ascribed to the initiation of recrystallization which was observed in figure 11.

Actually, this small increase in the grain size provokes only a small degree of the microhardness drop (between 4 and 6 %). The small increase of the grain size accompanied with the small decrease of

**Table I.** The effect of high-temperature exposure at 800 °C for 5 h on the grain size and microhardness on Cu-Al<sub>2</sub>O<sub>3</sub> composite compacts processed from 5- and 20 h-milled powders and electrolytic copper\*.

*Tabla I. Efecto de exposición a 800 °C durante 5 h sobre el tamaño de de grano y sobre la micro dureza del compuesto compacto Cu-Al<sub>2</sub>O<sub>3</sub> procesado durante 5- y 20 h- a partir de polvos molidos y cobre electrolítico\*.*

Properties	Before exposure		After exposure		Increase of grain size,%		Decrease of microhardness, %	
	Milling time, h		Milling time, h					
	5	20	5	20	5	20	5	20
Grain size, nm	75 <sup>†</sup>	45	82	49	8.5	8.2		
	90*	54*	121*	104*	34*	93*		
Microhardness, HV	255	292	239	279			6.2	4.5
	96*	260*	34*	73*			64.5*	72*

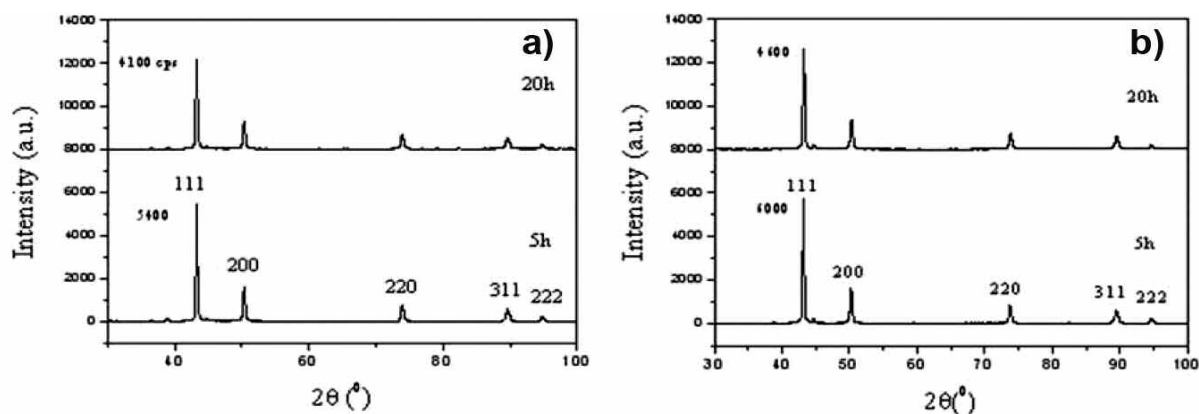
<sup>†</sup> Calculated values of grain size are inserted in Table I.

microhardness during exposure at 800 °C is a clear indication of the high thermal stability of this composite.

The measured densities of composite compacts processed from powders milled for 5 h and 20 h were 93.1 % and 94.5 % (7.74 and 7.85 gcm<sup>-3</sup>, respectively) of theoretical density (8.46 gcm<sup>-3</sup>). This indicates that the densification by hot-pressing of milled prealloyed powder was not completed. Hot-extrusion seems to be a common method of compaction because the measured density of Cu-based extruded composites is greater than 99.3 % [15].

The measurements of electrical conductivity of composite compacts in function of the time of milling are summarized in table II.

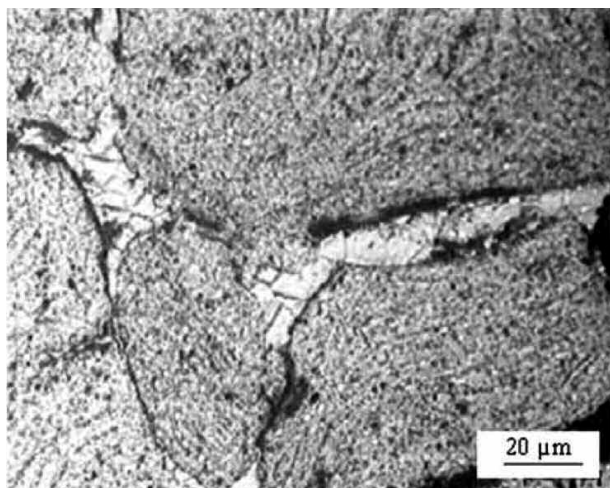
No significant change in electrical conductivity was detected with increase of milling time. The electrical conductivity of composite compacts processed from powders milled for 5 and 20 h was 37 and 38 % IACS, respectively. It should be noted that the conductivity requirement for the copper-based alloys for higher temperature applications is 50 % IACS [17]. On the other side, electrical conductivity of commercially available Cu-Al<sub>2</sub>O<sub>3</sub> based composites



**Figure 10.** The XRD of compacts before (a) and after (b) high temperature exposure at 800 °C.

*Figura 10. XRD de los compuestos antes (a) y después (b) de la exposición a temperaturas de 800 °C.*





**Figure 11.** Optical microscope. Microstructure of 20 h-milled compact after high temperature exposure at 800 °C.

*Figura 11. Microscopio óptico. Microestructura del compuesto molido durante 20h, después de la exposición a temperaturas de 800 °C.*

ranges between 78 and 92 % IACS due to the lower content of  $\text{Al}_2\text{O}_3$  (less than 3 wt. %) [18]. The low electrical conductivity measured in this work, is not only the consequence of higher content of  $\text{Al}_2\text{O}_3$  particles in the copper matrix, but some other parameters should be considered. The low electrical conductivity may be prescribed to the inadequate density of Cu- $\text{Al}_2\text{O}_3$  composite. Also, the residual aluminium (remained in the solid solution) may significantly reduce electrical conductivity (probably much more than the  $\text{Al}_2\text{O}_3$ ). Although our results indicate that the whole amount of aluminium was oxidized, the negative effect of some remained quantity in the solid solution cannot be excluded.

The effect of high-temperature exposure on the electrical conductivity of composite processed from 5 and 20 h milled powders is summarized in table III.

Somewhat higher electrical conductivity after exposure is related to the slight increase in the grain size as it can be seen in table I [19].

#### 4. CONCLUSIONS

The present study leads to the following conclusions:

- The decrease of Cu-3 wt. % Al lattice parameter with milling time, is the result of oxidation of aluminium which precipitates from prealloyed copper forming a fine dispersion of  $\text{Al}_2\text{O}_3$  particles. Assuming that the complete amount

**Table II.** The effect of milling time on electrical conductivity of Cu- $\text{Al}_2\text{O}_3$  composite

*Tabla II. Efecto del tiempo de molienda sobre la conductividad térmica del compuesto Cu- $\text{Al}_2\text{O}_3$*

Electrical conductivity, % IACS				
Milling time, h				
0*	3	5	10	20
27	34.5	37	37.5	38

\* Composite processed from as-received and non-milled powders

**Table III.** The effect of high-temperature exposure at 800 °C for 5 h on electrical conductivity of Cu- $\text{Al}_2\text{O}_3$  composite processed from 5 and 20 h milled powders.

*Tabla III. Efecto de la exposición a 800 °C durante 5 h sobre la conductividad eléctrica del compuesto Cu- $\text{Al}_2\text{O}_3$  procesado a partir de polvo molido durante 5 y 20 h.*

Electrical conductivity, % IACS			
Before exposure		After exposure	
Milling time, h		Milling time, h	
5	20	5.0	20
37	38	40.3	44

of aluminium was oxidized, it was calculated that 5.6 wt.% of  $\text{Al}_2\text{O}_3$  was produced in the copper matrix by internal oxidation of 3 wt. % Al.

- The increase of microhardness of Cu- $\text{Al}_2\text{O}_3$  composite is a consequence of very small grain size formed during high-energy milling, although the effect of very fine  $\text{Al}_2\text{O}_3$  particles generated by internal oxidation must be taken into account. The effect of internal oxidation and high-energy milling on strengthening of Cu- $\text{Al}_2\text{O}_3$  composite was significant reflecting in almost threefold increase of the microhardness of composite compacts (292 HV) compared to compacts processed from the as-received Cu-3 wt. % Al powder (102 H).
- The small increase in the grain size (approximately 8 %) provokes only a small degree (between 4

and 6 %) of the microhardness drop indicating the high thermal stability of Cu-Al<sub>2</sub>O<sub>3</sub> composite during high-temperature exposure at 800 °C.

- The electrical conductivity of composite does not depend on the milling time and remained practically unchanged after high-temperature exposure. The values of electrical conductivity of composite processed from 5 and 20 h milled powders (37 and 38 % IACS, respectively) imply that the electrical conductivity depends not only on the amount of Al<sub>2</sub>O<sub>3</sub> particles, but the effect of some other parameters should be also considered.

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